

TITLEHIGH STRETCH RECOVERY NON-WOVEN FABRIC AND PROCESS  
FOR PREPARINGFIELD OF THE INVENTION

5           The present invention is concerned with a novel non-woven fabric characterized by high stretch recovery and a process for preparing said fabric by employing fibers of latent crimp.

BACKGROUND OF THE INVENTION

10           Non-woven fabrics made from thermoplastic synthetic fibers are well-known in the art and in widespread commercial use under such trade names as Tyvek® and Sontara®, both available from the DuPont Company, and the like.

15           Long sought in non-wovens is a high bulk textile like product having high stretchability combined with high stretch recovery. Such fabrics are known collectively as "stretch non-wovens." Numerous approaches have been taken to preparing stretch non-wovens.

20           One approach to producing stretch non-wovens has been to employ crimped staple fibers, wherein the entanglement of crimped fibers provides the cohesion and recovery needed in a stretch non-woven. In some instances in the art, a crimped fiber is formed into a mat and interlaced by use of an air or water jet. In other cases in the art, a straight or substantially straight fiber having latent crimp is first formed into a mat after which the latent crimp is realized by heating, thereby creating a "self-entangled" structure. However, the stretch non-wovens of the art lack the toughness, defined as the product of tensile modulus and ultimate elongation, and the density to be useful in many textile and industrial applications. It is the toughness of the fabric which determines its "stretchiness", that is the amount it can be stretched and the power of its recovery. The stretch-nonwovens of the art are limited to low density, or, to use a term of art, high loft fabrics with very low stretchiness.

30           Aranaga et al, Japanese Kokai Heisei 11-158733 discloses a wet-lay process for forming a non-woven from a bicomponent fiber of polyethylene terephthalate/polypropylene terephthalate having latent crimp. The resulting non-woven is said to have a high percentage of recovery. The wet-lay process includes the step of hydroentangling the fibers in order to provide sufficient entanglement prior to developing the crimp. The non-woven fabrics thereby produced are characterized by a basis weight of 30 g/m<sup>2</sup> in a 0.3 mm thick fabric. It is recognized in

Aranaga and elsewhere that high recovery crimped fibers have potential for preparation of high value non-wovens. However, the challenge, clearly recognized in Aranaga, is that fibers having high retractive force tend to crimp independently resulting in an increase in fabric bulkiness at the expense of a decrease in entanglement formation. It is also often associated with fabric breakage during shrinkage. The result is typically a high bulk fabric suitable for non-load-bearing applications, and the full potential of the high recovery fiber is not realized.

Furukawa, U.S. Patent 4,469,540, discloses highly bulky non-wovens having bulk density  $<0.020 \text{ g/cm}^3$  formed from crimped polyolefin bicomponent staple fibers. Crimp frequency of more than 12 crimps per inch is said to be detrimental to web formation because it makes the web density excessively high. The structure according to Furukawa is said to be "melt-adhered" by thermal bonding and said melt-adherence, rather than fiber entanglement, is said to be the principal mechanism by which the structure is held together.

Stokes et al, WO 00/18995, discloses a method for forming compression resistant bulky non-wovens from crimped bicomponent polyolefin fibers followed by cross-linking to maintain resiliency.

Terakawa et al, EP 0 391 260 B1, discloses a process for making non-woven fabrics from continuous bicomponent polyolefin fibers in which the as spun yarn bundles having latent crimp are air-entangled, heated to a temperature at which the crimp develops, and heated to a temperature to cause bonding at the cross-over points. Non-woven fabrics according to Terakawa have densities below  $0.030 \text{ g/cm}^3$ .

Shawver et al, U.S. Patent 5,540,976, discloses stretch non-wovens made from spun-bonded material laminated to an elastomeric inner layer sheet.

Pike et al, U.S. Patent 5,418,045, discloses formation of a non-woven web from continuous multicomponent thermoplastic fibers having latent crimp, especially polyolefins, and crimped to varying degrees in air prior to formation of a thermally bonded non-woven web.

#### SUMMARY OF THE INVENTION

The present invention provides for a non-woven fabric comprising a plurality of entangled helically crimped asymmetric bicomponent fibers comprising a first crystallizable polyester component and a second crystallizable polyester component, said first crystallizable polyester component exhibiting a lower rate of crystallization than said second

crystallizable polyester component, said fibers being characterized by a denier range of 0.5 to 6 denier, said fibers exhibiting at least 50 crimps per inch with a crimp radius of curvature of 0.2 mm or less, and wherein said fibers are preponderantly entangled with one another, and wherein further  
5 said fibers are preponderantly oriented in a well-defined plane said non-woven fabric being characterized by a bulk density of 0.2-0.4 g/cm<sup>3</sup>.

The present invention further provides for a process for forming a non-woven fabric, the process comprising disposing a plurality of asymmetric bicomponent fibers having latent crimp in a planar array of  
10 overlapping fibers, said fibers being preponderantly oriented in the plane thereof, disposing said planar array between two constraining surfaces; heating said planar array to develop at least a portion of said latent crimp with the proviso that during at least a portion of said heating, said non-woven structure is in constraining contact with said constraining surfaces.

#### 15 BRIEF DESCRIPTION OF THE DRAWING

Figure 1 depicts the disposition of the wet fibrous mat precursor and cloth support on the drying drum of the Williams Pulp Testing Apparatus.

#### DETAILED DESCRIPTION OF THE INVENTION

It is known in the art to prepare non-woven fabrics by preparing a  
20 planar array, typically a fibrous mat, of bicomponent fibers having latent crimp followed by heating to develop the crimp, thereby entangling the fibers to produce a stable non-woven fabric structure. During the crimp-development step, the fibrous mats or other structures undergo shrinkage in the plane with concomitant expansion in the direction normal to the  
25 plane. As a result, the non-woven fabrics of the art are in general of quite low bulk density, well below 0.1 g/cm<sup>3</sup>. As a consequence, they have limited utility in many textile applications which require high toughness or high stretch recovery.

The present invention is based upon the discovery that  
30 entanglement formation among high crimp, high recovery fibers can be incited through a careful control of the fabric expansion in the direction normal to the plane of the fabric during the process of crimp development. In processes of the art crimp development normally leads to extensive shrinkage in the plane with concomitant expansion in the direction normal  
35 to the plane. .

In the process of the present invention, in a first step a plurality of asymmetric bicomponent fibers having latent crimp is disposed to form a planar array of said fibers, said fibers being preponderantly oriented in the

plane thereof, wherein a preponderance of said fibers are in cross-wise contact with at least one other said fiber. Said planar array in a preferred embodiment is referred to herein as a "fibrous mat preform" to indicate a non-woven fabric structure prior to being subject to a second step of the process of the invention, the crimp development step. The crimp development step is performed by heating the planar array to develop the crimp while the planar array is disposed between two constraining surfaces oriented at least approximately parallel to the plane of the planar array. The separation of the two constraining surfaces is adjusted so that during at least some portion of the crimp development step the expansion of the planar array in the direction normal to the planar surface is constrained by simultaneous contact with both constraining surfaces. This contact during the expansion step introduces a compressive force on the shrinking array. The resulting non-woven fabric exhibits higher density and more robust physical properties than a non-woven made from a comparable starting material which is produced without constraining the expansion in the direction normal to the planar surface.

Bicomponent fibers suitable for the process of the present invention are fibers comprising two polymers, preferably of the same generic family, present in two continuous phases contiguous with one another, which two phases exhibit differential shrinkage thereby enabling the development of a helical crimp upon shrinkage. The phases may be arranged in a side-by-side or asymmetric sheath-core arrangement. Side-by-side is preferred. Suitable bicomponent fibers include but are not limited to bicomponent polyesters, bicomponent polyamides, and bicomponent polyolefins. Polyesters are preferred. Copolymers of polymers within these broad classes are included therein. Preferred species of bicomponent fibers among polyesters include polyethylene terephthalate (PET)/polypropylene terephthalate (PPT), PET/polybutylene terephthalate, (PBT), and PPT/PBT, preferred is PET/PPT.

Also encompassed by the term "bicomponent," are fibers sometimes referred to as biconstituent—that is comprising polymers from different families, such a polyamide and polyester. However, biconstituent fibers are less preferred.

For the purposes of this invention, a fiber having latent crimp is a fiber which has the inherent capacity to develop additional crimping by exploiting differences in shrinkage behavior of the two components, typically by heating at least one of the components above its glass

transition temperature. A latent crimp fiber may exhibit some crimps, or none. In the practice of the present invention, the latent crimp fiber is preferably flat, without crimps.

5 The inventors hereof have discovered that when crimp development is performed under conditions in which the expansion of the fabric in the direction normal to the plane thereof is constrained, as for example by performing the crimp development step with the fabric positioned between two metal plates parallel to the plane of the fibrous mat preform, the effect is to cause the fabric to undergo significant densification, and the  
10 individual fibers to become more highly entangled. The result is a denser, tougher fabric with improved stretch recovery over a fabric prepared by the prior art method starting with an identical fibrous mat preform.

The degree of densification and the properties which can be achieved will depend upon the crimp contraction of the particular type of  
15 fiber employed in the invention, as well as the starting bulk density of the planar array, the degree to which the fibers overlap one another, and the distance of separation between the constraining surfaces. Other things being equal, higher crimp contraction, greater degree of overlap among the fibers, higher starting bulk density, and a narrower gap between  
20 constraining surfaces, (up to the point where the fiber can no longer slip, and crimping is significantly inhibited) are all associated with a higher density the non-woven product, higher toughness, and higher the stretch recovery.

The benefits of the process of the invention are quite general, and  
25 may be applied to preparation of non-wovens of essentially any composition provided that bicomponent fibers with latent crimp are employed in a planar array of overlapping fibers.

The fibers employed in the invention may be in the form of continuous or long fibers, or they may be staple fibers. Continuous fibers  
30 may be spun in the form of multifilament yarns but are preferably deposited as individual fibers to make up the fibrous mat precursor. Staple fibers are preferred, with fibers in the length range of 3 to 25 mm preferred, and fiber deniers in the range of 0.5 to 6 denier per filament (dpf). In a preferred embodiment, the fibers are staple bicomponent fibers  
35 of PET and PPT in the concentration ratio of 70:30 to 30:70 respectively, preferably 60:40 to 40:60 respectively.

In a more preferred embodiment, the fibers employed in the invention are uncrimped staple PET/PPT bicomponent fibers having a

latent crimp contraction of at least 40%, preferably 70-80%. Both PET and PPT are crystallizable polymers. However, PPT exhibits a higher crystallization rate than PET.

PET/PPT fibers preferred for use in the present invention may be prepared by combining a melt stream of PET having an intrinsic viscosity (I.V.) of, 0.4 to 0.8, preferably 0.5 to 0.6 with a melt stream of PPT having an I.V. of 0.8 to 1.5, preferably 0.9 to 1.0, and feeding the combined streams to a multihole spinneret wherefrom it is extruded at a temperature of 260 to 285°C, preferably 265° to 270°C. The extrudate is collected and quenched, and then wound up without a drawing step. The spinning speed is in the range of 1900 to 3500 m/min, with 2000 to 3000 m/min preferred. After spinning, the yarns are subject to cutting to lengths of 3 to 25 mm, with lengths of 20 to 25 mm preferred. I.V. is as determined in p-chlorophenol at 25°C.

The practitioner hereof will understand that the specific value of the spinning speed which will provide the desired fiber properties will depend upon the specific choices of polymers employed, the specifics of spinning temperature, fiber diameter, and type of quenching. The practitioner hereof will further understand that the spinning speed range suitable for PET/PPT fibers will be different from the spinning speed range suitable for other compositions. For example, it has been found that for PET/PBT fibers the operating range is about 1700 to 3200 m/min, with 1800 to 3000 m/min preferred; and for PPT/PBT fibers the operating range is it is 600 to 2000 m/min, with 800 to 1600 m/min preferred.

The staple fiber yarns so prepared are then dispersed in water with the aid of a surfactant at solids content in the range of 0.05 g/l to 1 g/l, preferably 0.25 g/l to 0.75 g/l. The dispersion is agitated without causing turbulence to obtain a homogeneous, well-separated mixture of fibers. The fiber dispersion is then deposited upon a porous substrate, the excess water drained out, and the resulting mat of fibers is dried at a temperature below 70°C, preferably ca. 40°C. Preferably drying is achieved while blotting the surface.

Other means and media for dispersing the fibers may be employed. The fibers may be dispersed using liquids besides water so long as the liquids are essentially inert. Or the fibers can be dispersed in gaseous media such as air, or in super-critical CO<sub>2</sub>. However, the water dispersion method, which employs techniques similar to that of the well-known method for making paper, is most convenient, and therefore preferred.

In an alternative embodiment of the present invention, the fibers are in the form of continuous multi-filament yarns having latent crimp. When such continuous filaments are employed a high degree of filament separation must take place in order to form the planar array or fibrous mat precursor suitable for the process of the invention. One way to achieve that filament separation is to impose an electrostatic charge on the yarn bundle. The moving multifilament yarn bundle is charged electrostatically to a potential sufficient to separate each filament from adjacent filaments, and then, while thus separated, the filaments are collected as a random nonwoven web. The preferred yarn possesses zero twist or crimp in order to effect maximum separation of the filaments. A minimum level of charge is 30,000 electrostatic units (esu). Charging is accomplished while the filaments are under sufficient tension so that they do not separate until such tension is released, i.e., after they have been urged toward the receiving surface upon which the planar array is to be formed, whereupon they immediately separate. The filaments may be charged by a corona discharge, by triboelectric contact, by field charging, or other suitable methods. In one embodiment, freshly formed synthetic organic filaments formed according to the process of the invention while still above their solidification temperature are charged by passing through a high intensity electric field. Suitable apparatus and detailed procedures for imposing electrical charge on yarn bundles suitable for the practice of the present invention are described in U.S. Patent 3,338,992. The filaments constituting the yarns so treated are laid down to form an overlapping pattern to create a planar array or fibrous mat precursor wherein the filaments are preponderantly oriented in the plane thereof.

The planar array of continuous fibers so prepared is then conveyed in a continuous or batch continuous manner to a heating zone wherein the latent crimp is developed, and the non-woven fabric of the present invention is produced. While many methods of providing heating are known in the art, any method suitable for the practice of the present invention will necessarily involve constraining expansion in the direction normal to the plane of the precursor fabric. Such method will include such batch type methods as a stationery heated zone between two plates, or such continuous process methods as calendering, or heated conveyors. The method by which the desired crimp is realized is not critical so long as it meets the proviso that during at least a portion of said heating, said non-woven structure is in constraining contact with the constraining surfaces.

Heating to develop the latent crimp of the fibers in the non-woven structure so produced may be accomplished using a variety of methods such as would be well within the purview of one of ordinary skill in the art. It is found in the practice of the invention that satisfactory results are  
5 achieved by heating: (i) in hot air as in a hot air oven with forced convective flow at a temperature above 80°C and preferably around 120°C or, (ii) in water at a temperature around 95°C. In the typical practice of the process of the invention crimp development occurs within a few seconds after the indicated thermal exposure. Hot air is the preferred heating  
10 medium when the distance between constraining surfaces exceeds about 2 mm. It is found in the practice of the process of the invention that hot water heating of samples thicker than ca. 2 mm often leads to sample fracture.

In a further embodiment, an objective of the present invention is to  
15 provide entangled nonwoven fabrics with high toughness and controlled properties ranging from high-stretch/ low-stiffness to high-stiffness/low-stretch. This is obtained through (i) using fibers with high latent crimp and (ii) restraining expansion of fabric thickness during thermally induced area shrinkage according to the process of the invention as herein described.  
20 Area shrinkage during crimp development is an indicator of the extent of crimp development.

The non-woven fabric of the invention has particularly desirable properties of toughness, defined as the product of the initial Young's modulus multiplied by the ultimate stretch thereof. When a non-woven  
25 fabric is prepared according to a preferred embodiment of the process of the invention, a fabric is produced which exhibits initial Young's modulus values ranging from 1.2 to 12 MPa and up to 150% ultimate stretch. Preferred embodiments of the non-woven fabric of the invention provide combinations of ultimate stretch and tensile modulus ranging from ca. 30%  
30 and 6 MPa, respectively, to 100% and 1.8 MPa respectively in fabrics having bulk densities of 0.20-0.28 g/cm<sup>3</sup>.

The non-woven fabric of the present invention comprises a plurality of entangled helically crimped side by side bicomponent fibers comprising a first crystallizable polyester component and a second crystallizable  
35 polyester component, said first crystallizable polyester component exhibiting a lower rate of crystallization than said second crystallizable polyester component, said fibers being characterized by a denier range of 0.5 to 6 dpf, said fibers exhibiting at least 50 crimps per inch with a crimp



radius of curvature of 0.2 mm or less, and wherein said fibers are preponderantly entangled with one another, and wherein further said fibers are preponderantly oriented in a well-defined plane said non-woven fabric being characterized by a bulk density of 0.2-0.4 g/cm<sup>3</sup>.

- 5           Density of the non-woven fabric of the invention is determined by cutting a specimen of known area, determining the thickness thereof and the weight thereof, and computing the density according to the formula:

$$\rho \text{ (density)} = \text{wt (g)} / (\text{area (cm}^2\text{)} \times \text{thickness (cm)})$$

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The non-woven fabric of the present invention is readily compressed, and present many interstices between fibers. Obtaining an accurate measurement of thickness in order to make an accurate determination of density is therefore problematical. Normally, thickness of objects such as  
15   films and fabrics is determined in the art by use of contact thickness gauge, in which the specimen to be measured is positioned between a fixed anvil and a vertically displaceable foot attached to some means for indicating thickness. If the vertically displaceable foot has too narrow a cross-section, it may slip between adjacent fibers, thereby providing an  
20   erroneously low thickness reading. If the vertically displaceable foot exerts excessive pressure on the area being measured, it may result in compression of the fabric, again resulting in an erroneously low thickness reading.

- In order to avoid these pitfalls, thickness measurement should be  
25   performed using a thickness gauge having a vertically displaceable foot with a flat specimen contact surface of circular cross-section of a diameter of at least 0.5 cm, and exerting a total force of no greater than 95 g. Gauge precision should be at least  $\pm 0.0005$  cm. While any instrument which meets those limitations is suitable for determining the thickness of  
30   the sample according to the invention, several commercially available instruments are available which will suffice. One such instrument found to be suitable for the thickness determination according to the invention is the Model PT223 Federal (Providence, RI) C2I Comparator gauge provided with a Model PT223 contact foot mounted on a Model 35B-8-R-1 stand.  
35   The thickness measurement should represent an average of at least 3 readings taken at different points on the specimen.

          Weight is determined on a laboratory balance having a precision of at least 0.0001 g.

The bicomponent fibers suitable for use in the non-woven fabric of the invention are preferably side-by-side bicomponent fibers selected from the group consisting of PET/PPT, PET/PBT, and PPT/PBT fibers. In the preceding group, said first crystallizable polyester component is listed first, and the second crystallizable component is listed second. In other words, the polymers are listed with the slower to crystallize polymer listed first. Most preferably, said bicomponent fiber is a side-by-side bicomponent fiber of PET/PPT.

PET/PPT fibers preferred for use in the non-woven fabric of the invention may be prepared by combining a melt stream of PET having an intrinsic viscosity (I.V.) of 0.5 to 1.2, preferably 0.7 to 0.9, with a melt stream of PPT having an I.V. of 0.8 dl/g to 1.5 dl/g preferably 0.9 to 1, and feeding the combined streams to a multihole spinneret wherefrom is extruded from each whole thereof a single bicomponent strand at a temperature of 265° to 285°C, preferably 265° to 270°C. The extrudate is collected and quenched, and then wound up without a drawing step. Wind-up speed, which in this case is synonymous with spinning speed, is in the range of 2000 to 3500 m/min, with 2500 to 3000 m/min preferred. After spinning, the yarns are subject to cutting to lengths of 3 to 25 mm, with lengths of 25 mm preferred. I.V. is as determined in p-chlorophenol at 25°C.

The preferred fibers so prepared are then processed according to the process hereof already described, resulting in the non-woven fabric of the invention.

The unusually high density and high recovery power of the non-woven fabrics of the present invention make them useful in areas such as fine particle filtration and protective applications. Nonwovens are ideal candidates for industrial dust removal applications as their random arrangement of fibers allows a rapid distribution of the carrier phase into individual currents. The vast majority of nonwovens are needle punched and usually known as "needle felts" with densities around 0.2 g/cm<sup>3</sup>. At same density, the fabric of the present invention is expected to have superior filtration efficiency as it readily leads to a 3-dimensional random arrangement of the fibers. The latter greatly facilitates the build-up of "dust bridges" which are crucial in order to rapidly reduce the penetration of dust to extremely low levels.

In another embodiment, the bicomponent fibers suitable for use in the present invention may be combined, preferably during the lay-down of

the fibrous mat precursor, with polyaramid fibers, such as Nomex® or Kevlar® fibers available from the DuPont Company, to enhance the strength, thermal resistance and puncture resistance of the resulting fabric. In this embodiment, the polyaramid staple fibers, which in general do not exhibit thermal shrinkage, are intermixed with the bicomponent fibers suitable for the practice of the invention, to form the fibrous mat precursor. During crimp development according to the process of the invention, the polyaramid fiber is subject to twisting and entanglement with the crimped bicomponent fibers forming a highly reinforced network of entangled fibers, tightly binding the polyaramid fibers to the non-woven fabric. This intimate bonding can be achieved without the use of binding agents as are commonly employed in making blends involving polyaramids.

Surface modifiers and additives can easily be incorporated into the non-woven fabric of the invention in order to provide, for example, antimicrobial and fire retardance properties. Other polymeric fibers which do or do not exhibit latent crimp can also be easily incorporated through a blending with the bicomponent fiber in the practice of the invention, particularly by combining the fibers in the water slurry stage of the preferred embodiment of the invention. The addition of short polyaramid fibers could, for example, be considered to improve fire retardance and abrasion resistance.

The present invention is further described but not limited to the following specific embodiments.

#### EXAMPLES

The fiber employed in the following examples was a bicomponent side-by-side yarn made of a 50/50 ratio of PET (Crystar 4415, IV=0.54+/-0.02) and PPT (CIDU, IV=1.04+/-0.03). The polymers were melt spun through a 34-hole spinneret at 265-270°C according to standard procedures in the art and employing an extrusion block and spinneret pack standard and well-known in the art for preparing side-by-side bicomponent fibers, as described in Evans et al, U.S. Patent US 3,671,379.

The extrudate was cooled by passing through a cross-flow quench zone 72 inches long with a flow of room temperature quench air moving at right angles to the yarn path at approximately 10 m/min. It was then wound up, without any separate draw stage, at the speed indicated in the specific example. The resulting yarn was straight, that is, it exhibited no visible crimp.

After spinning, the yarn was rewound in lengths of 90 m each on a small motor-driven skeiner having diameter of 11 cm. Each skein was then cut with scissors into short fiber floc with length indicated in the specific example. A solution of 5 grams of F-98 Prill, a surfactant available from BASF Corp., Mt. Olive, NJ, in 2 liters of water was poured into the reservoir of a Williams Standard Pulp Testing Apparatus having dimensions 28 cm x 28 cm x 28 cm. Additional cold water was added to fill the reservoir. 6 g of floc was then gently disbursed in the pulp apparatus using a spatula and the aqueous slurry was agitated for about 30 seconds using a hand-held agitator consisting of a wide steel plate with holes in it. Great care has to be provided to avoid turbulence which inevitably lead to flocculation. The water was then drained out thereby depositing a fiber mat on the porous cloth at the bottom of the reservoir.

The cloth and the fiber mat were then transferred onto a sheet drier also manufactured by Williams Apparatus, Watertown, NY. The sheet drier, shown in Figure 1, consisted of a porous metal drum, 1, upon half or less of the circumference of which, the cloth and fiber mat, 2, were disposed. To secure the mat into place, a canvas sheet, 3, affixed at one end, 4, upon the surface of the drum, is placed over the cloth and fiber mat, 2, and tightened by rotating a knob, 5, to which the other end of the canvas sheet is attached. By rotating the knob manually to the desired degree of tightness, the pressure exerted on the cloth and fiber mat by the canvas sheet may be varied over a wide, although somewhat subjective, range.

The mat was dried at 35°C-40°C. The drying was performed under slight pressure which was obtained by tightening the canvas covering the sheet by about two turns of the knob. After an hour drying time, the mat was removed and cut into square 3" x 3" samples. Each sample was then individually inserted between two 16 cm x 21 cm x 0.7 cm teflon-coated aluminum plates weighting 554 g each. The distance between the plates was controlled by the insertion of shims with thickness varying from 0.25 to 4 mm. The composite (2 plates + sample + shims) was then heated by hot air at 120°C for about 30 minutes.

All the mechanical testing was done using a table top Instron Tensile Tester, Model 1123. The machine had been upgraded with MTS Renew Package that contains MTS Testworks software version 4.0.

The crimp contraction (CC) of the yarn to be employed in the process of the invention was evaluated as follows: A 4" piece of yarn was

heated in hot air at 120°C for 30 minutes. That piece was then hung on a hook from its midpoint, thereby forming a loop, and its two ends were taped together. Denoting by  $L_{1.5}$  the length of the yarn under a 1.5 mg/d load attached to its taped ends and by  $L_{100}$  that under a 100 mg/d load, the crimp contraction was calculated as  $CC (\%) = [(L_{100} - L_{1.5}) / L_{100}] \times 100$ . Three specimens were averaged to get the result.

To determine the number of crimps per inch (CPI), a separate specimen of yarn was heated as in the crimp contraction test. It was then sandwiched between two glass plates and examined using a stereoscope with top lighting at a magnification of ~16X. The CPI number was obtained by calculating the number of peaks on one side of the fiber axis over a section having end-to-end distance equal to one inch.

Density was determined by measuring thickness of a 3"x3" (7.62 cm x 7.62 cm) square specimen using a Federer C2I Comparator gauge, mounted on a stand (Model 35B-8-R-1). The comparator has a flat contact point Model PT223 exerting a total force of  $93 \pm 2$  g, and a foot diameter of 0.18" (0.46 cm). Thickness was an average of five determinations at different places on the specimen.

The weight of the fabric specimen was determined using a Mettler 8200 balance having a precision 0.0001 g.

Prior to heat treatment, a typical wet-laid sheet made of 1/4" floc has thicknesses 0.28 +/- 0.02 mm.

Area shrinkage was measured for the whole sample using the relation  $100 \times (A_{\text{before}} - A_{\text{after}}) / A_{\text{before}}$  in which  $A_{\text{before}}$  and  $A_{\text{after}}$  represent the area of the square sample before and after shrinkage.

#### Example 1:

A 34 filament 50/50 PET/PPT bicomponent yarn having 5.9 denier per filament was spun at a speed of 3030 m/min, according to the process described hereinabove. The yarn bundle exhibited a tensile strength of 1.6 grams/denier. The yarn so prepared was cut into 1" floc with scissors and processed in the a Williams Standard Pulp Testing Apparatus using the method described hereinabove, with hot air as the heating agent. The resulting crimped yarn had the following characteristics: crimp contraction  $CC=74\%$ , the number of crimps per inch was  $CPI=58$ , with average radius of curvature of 0.13 mm. The characteristics of the fabric for different shim thicknesses are shown in Table I. Each value was an average over two samples. The product of the modulus by the elongation (referred to as toughness) had a constant value around 2.

Table I

Shims (mm)	Area Shrinkage (%)	Surface Density (g/m <sup>2</sup> )	Modulus (g/d)	Stretch at Maximum Load (%)	Modulus x Stretch (g/d x. %)	Density (g/cm <sup>3</sup> )
0.25	44	226	0.055	35	1.9	0.28
0.50	58	179	0.045	50	2.2	0.24
1	67	350	0.035	70	2.4	0.23
2	79	533	0.022	88	1.9	0.23
4	84	504	0.014	95	1.4	0.20

Example 2

- 5           The materials and procedures of Example 1 were repeated with the exception that, prior to dispersion in the Williams Standard Pulp Apparatus, the 1" floc was completely wetted out in a 1% w/w solution of Polyethylene Oxide (Mw=900,000) in water. The results are presented in Table II.

10

Table II

Shims (mm)	Area Shrinkage (%)	Surface Density (g/m <sup>2</sup> )	Modulus (g/d)	Stretch at Maximum Load (%)	Modulus x Stretch (g/d x. %)	Density (g/cm <sup>3</sup> )
0.25	48	163	0.053	34	1.8	0.26
0.50	47	180	0.046	44	2.0	0.24
1	72	270	0.029	58	1.7	0.24
2	79	422	0.017	93	1.6	0.22
4	86	552	0.012	120	1.5	0.22

Example 3:

- 15           The materials and procedures of Example 1 were repeated with the exception that the fiber was cut into 1/4" floc. The results are presented in Table III.

Table III

Shims (mm)	Area Shrinkage (%)	Surface Density (g/m <sup>2</sup> )	Modulus (g/d)	Stretch at Maximum Load (%)	Modulus x Stretch (g/d. %)	Density (C2I gauge) (g/cm <sup>3</sup> )
0.25	49	144	0.038	28	1.1	0.24
0.50	67	204	0.024	48	1.2	0.24
1	75	286	0.022	64	1.4	0.24
2	81	348	0.010	90	0.9	0.21
4	86	546	0.013	82	1.1	0.23

Comparative Example 1:

- The methods and materials of Example 3 were repeated except that the yarn was spun at 1850 m/min resulting in a 4.7 denier per filament yarn with a tensile strength of about 1.17g/d. Upon development of its latent crimp following the methods outlined hereinabove, the yarn had the following characteristics: crimp contraction CC=0.42; number of crimps per inch CPI=17 with average radius of curvature around 1.1 mm. The results are presented in Table IV. The floc in this example was 0.64 cm in length.

Table IV

Shims (mm)	Area Shrinkage (%)	Surface Density (g/m <sup>2</sup> )	Modulus (g/d)	Stretch at Maximum Load (%)	Modulus x Stretch (g/d. %)	Density (g/cm <sup>3</sup> )
0.25	0	68	0.10	3	0.3	0.18
0.50	0	70	0.09	4	0.4	0.19
1	8	71	0.02	7	0.2	0.19
2	28	92	0.007	19	0.2	0.20
4	49	145	0.004	40	0.2	0.19

Comparative Example 2:

- The materials and procedures of Example 1 were repeated except that the floc length was 51 mm. The results are presented in Table V.

Table V

Shims (mm)	Area Shrinkage (%)	Surface Density (g/m <sup>2</sup> )	Modulus (g/d)	Stretch at Maximum Load (%)	Modulus x Stretch (g/d . %)
0.50	59	171	0.045	55	2.5
1	76	260	0.017	85	1.5
2	82	385	0.012	139	1.7
4	87	692	0.010	152	1.5

Comparative Example 3

5 This example illustrates that, at drying temperatures higher than 60°C, fibers constrained from shrinking undergo heat-setting, thereby erasing the latent crimp.

The materials and procedures of Example 3 were repeated with drying effected at various temperatures as shown in Table VI. However, unlike in Example 3, the canvas sheet covering the fibrous mat during  
 10 drying was tightened very considerably in order to exert a pressure on the fibrous mat and constrain the mobility of the fibers, thereby preventing shrinkage. The results are for a yarn spun at 3030 m/min, cut into 1/4" floc and subsequently heat treated in hot air using 2 mm shims.

Table VI

Drying Temperature (°C)	Area Shrinkage (%)	Surface Density (g/m <sup>2</sup> )	Modulus (g/d)	Stretch at Maximum Load (%)	Modulus x Stretch (g/d. %)
23	79	364	0.013	84	1.1
40	82	432	0.013	98	1.3
60	73	290	0.017	74	1.3
80	0	64	0.009	41	0.4

Comparative Example 4

The materials and procedures of Example 3 were repeated except that the crimp development stage was performed in 95°C water. The  
 20 results are presented in Table VII. The samples at large shim thickness >1 mm broke in the water.



**Table VII**

Shims (mm)	Area Shrinkage (%)	Surface Density (g/m <sup>2</sup> )	Modulus (g/d)	Stretch at Maximum Load (%)	Modulus x Stretch (g/d . %)	Density (g/cm <sup>3</sup> )
0.25	28	94	0.027	24	0.6	0.27
0.50	40	125	0.056	22	1.2	0.28
1	75	302	0.024	31	0.7	0.33
2	-	-	-	-	-	-
4	-	-	-	-	-	-

**Comparative Example 5**

Same as Comparative Example 4 but, for a yarn spun at  
5 1850m/min. The results are presented in Table VIII.

**Table VIII**

Shims (mm)	Area Shrinkage (%)	Surface Density (g/m <sup>2</sup> )	Modulus (g/d)	Stretch at Maximum Load (%)	Modulus x Stretch (g/d . %)	Density (g/cm <sup>3</sup> )
0.25	0	64	0.23	3	0.7	0.20
0.50	0	73	0.038	5	0.2	0.23
1	11	76	0.029	9	0.3	0.20
2	48	132	0.011	23	0.3	0.24
4	76	323	0.011	37	0.4	0.20